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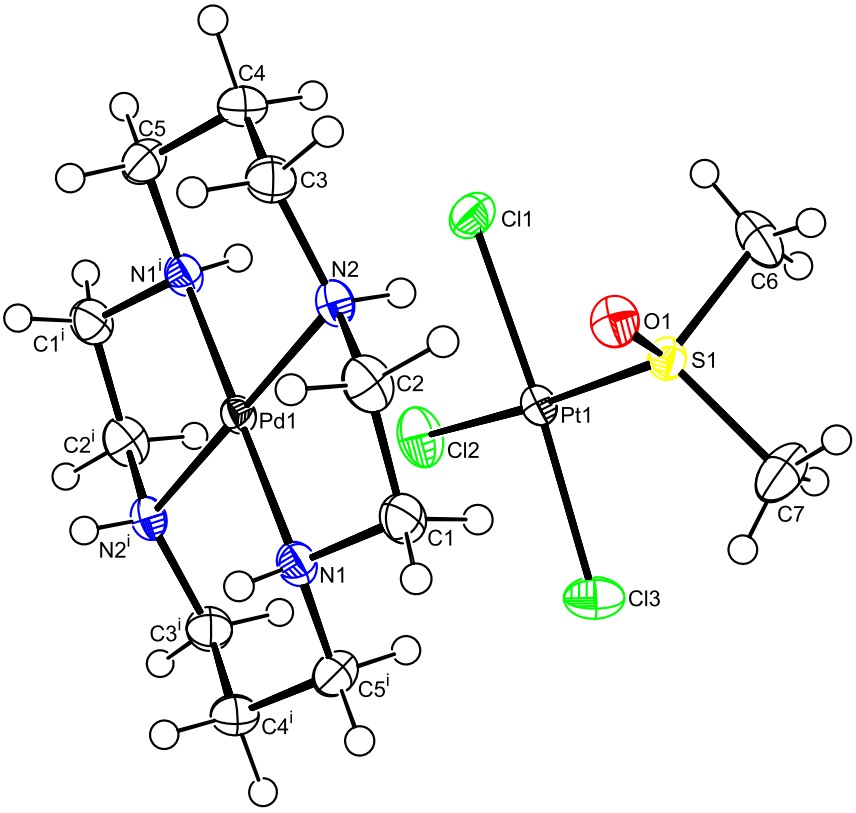
Crystal structure of (1,4,8,11-tetraazacyclotetradecane-κ^4N,N',N'',N''') palladium(II) bis[trichlorido(dimethyl sulfoxide-κS)platinate(II)], $C_{14}H_{36}Cl_6N_4O_2PdPt_2S_2$



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Abstract

$C_{14}H_{36}Cl_6N_4O_2PdPt_2S_2$, triclinic, $P\bar{1}$ (no. 2), a = 8.714(2) Å, b = 9.270(2) Å, c = 9.525(2) Å, α = 68.940(6)°, β = 87.786(7)°, γ = 77.327(7)°, V = 699.9(3) Å^3, Z = 1, $R_{gt}(F)$ = 0.0153, $wR_{ref}(F^2)$ = 0.0351, T = 223 K.

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The title crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Yellow Block
Size:	0.16 × 0.16 × 0.12 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	11.348 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω scans
θ_{max}, completeness:	28.4°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	24,206, 3,488, 0.042
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3,374
$N(param)_{refined}$:	214
Programs:	Bruker[1], SHELX[2], ORTEP-III[3], PLATON[4]

1 Source of materials

To a solution of [Pd(cyclam)]Cl_2 (cyclam = 1,4,8,11-tetraazacyclotetradecane; 0.0836 g, 0.221 mmol) in methanol

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Pd1	0.5000	0.0000	0.5000	0.01681 (6)
N1	0.6645 (3)	0.1309 (3)	0.4094 (3)	0.0214 (4)
H1	0.745 (3)	0.084 (3)	0.454 (3)	0.018 (7)*
N2	0.4303 (3)	0.1554 (3)	0.6092 (3)	0.0207 (4)
H2	0.356 (4)	0.217 (4)	0.556 (3)	0.022 (8)*
C1	0.6176 (3)	0.2815 (3)	0.4381 (4)	0.0271 (6)
H1A	0.540 (4)	0.350 (4)	0.360 (4)	0.047 (10)*
H1B	0.702 (4)	0.326 (3)	0.426 (3)	0.024 (8)*
C2	0.5572 (3)	0.2456 (3)	0.5943 (4)	0.0279 (6)
H2A	0.639 (4)	0.180 (4)	0.664 (4)	0.031 (8)*
H2B	0.514 (4)	0.344 (4)	0.615 (3)	0.030 (8)*
C3	0.3895 (3)	0.0833 (4)	0.7683 (3)	0.0276 (6)
H3A	0.360 (3)	0.168 (4)	0.806 (3)	0.027 (8)*
H3B	0.474 (4)	0.012 (4)	0.824 (4)	0.029 (8)*
C4	0.2603 (3)	−0.0070 (4)	0.7834 (3)	0.0286 (6)
H4A	0.171 (4)	0.055 (4)	0.724 (3)	0.024 (7)*
H4B	0.226 (3)	−0.038 (3)	0.888 (3)	0.024 (7)*
C5	0.3077 (3)	−0.1596 (3)	0.7517 (3)	0.0278 (6)
H5A	0.229 (4)	−0.218 (4)	0.781 (4)	0.034 (9)*
H5B	0.399 (4)	−0.225 (4)	0.806 (4)	0.032 (8)*
Pt1	0.17110 (2)	0.30101 (2)	0.19911 (2)	0.01987 (4)
Cl1	0.04200 (8)	0.19655 (8)	0.41570 (8)	0.03112 (14)
Cl2	0.15558 (10)	0.08719 (9)	0.13266 (10)	0.03947 (18)
Cl3	0.31339 (9)	0.39284 (10)	−0.01036 (8)	0.03681 (16)
S1	0.16601 (7)	0.50600 (7)	0.26631 (7)	0.02077 (12)
O1	0.2547 (2)	0.4741 (2)	0.4074 (2)	0.0263 (4)
C6	−0.0311 (4)	0.5964 (4)	0.2835 (4)	0.0345 (7)
H6A	−0.021 (4)	0.675 (4)	0.313 (4)	0.039 (9)*
H6B	−0.073 (4)	0.515 (4)	0.362 (4)	0.035 (9)*
H6C	−0.081 (4)	0.633 (4)	0.195 (4)	0.045 (10)*
C7	0.2237 (5)	0.6674 (4)	0.1240 (4)	0.0380 (7)
H7A	0.215 (4)	0.746 (5)	0.169 (4)	0.052 (11)*
H7B	0.156 (4)	0.692 (4)	0.043 (4)	0.047 (11)*
H7C	0.332 (5)	0.634 (5)	0.100 (5)	0.059 (12)*

(10 ml) was added $Na_2PtCl_4{\cdot}xH_2O$ (0.1003 g, 0.262 mmol in anhydrous basis) and stirred for 3 h at room temperature. The formed precipitate was separated by filtration, washed with methanol, H_2O and acetone, and dried at 60 °C, to give a pale yellow powder (0.0884 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from a dimethyl sulfoxide (DMSO) solution at 90 °C.

2 Experimental details

All H atoms were located from Fourier difference maps and refined isotropically: *d*(C–H) = 0.91(3)–0.99(3) Å and *d*(N–H) = 0.78(3), 0.81(3) Å in cyclam ligand and *d*(C–H) = 0.88(4)–0.97(4) Å in DMSO ligand. The highest peak (0.76 e Å^{-3}) and the deepest hole (–1.20 e Å^{-3}) in the difference Fourier map are located 0.89 Å and 0.77 Å from the atom Pt1.

3 Comment

The crystal structures of the related cyclam-Pd(II) complexes [Pd(cyclam)][M(CN)$_4$] (M = Pd, Pt, Ni)[5–7] have been determined previously.

The title compound consists of a cationic Pd(II) complex $[Pd(cyclam)]^{2+}$ and two anionic Pt(II) complexes $[PtCl_3(DMSO)]^-$. In the cationic complex, the Pd(II) ion is coordinated by four N atoms from the tetradentate cyclam ligand in a distorted square-planar coordination geometry and is located on an inversion center, and thus the asymmetric unit contains one half of the compound. The tight N–Pd–N chelating angles with 85.11(9)° and 94.89(9)° contribute the distortion of the square-plane. The Pd–N bond lengths are almost equal with *d*(Pd1–N1/2) = 2.046(2) and 2.042(2) Å. In the anionic complex, the Pt(II) ion is four-coordinated in a slightly distorted square-planar environment by three anionic Cl ligands and one S atom derived from a DMSO molecule. The Pt–Cl bond lengths are almost equal with *d*(Pt1–Cl1/2/3) = 2.3070(8), 2.3181(8) and 2.3009(8) Å. In the crystal structure, the complexes display weak inter- and intramolecular N–H···O, N–H···Cl and C–H···Cl hydrogen bonds with distances of 2.9876(7)–3.7272(9) Å between the donor and acceptor atoms, to stabilize the three-dimensional packing.[4]

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